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## MEMORANDUM

DATE: 17 November 1998

TO: David Bennett, WAM, U.S. EPA, Region X

FROM: Michelle Turner, Chemist, WESTON, Seattle  
Roger McGinnis, Senior Environmental Chemist, WESTON, Seattle

SUBJECT: Validation of Organotin Data  
Laboratory Batch: K9805457  
Site: Duwamish River

WORK ASSIGNMENT NO: 46-35-0JZZ

WORK ORDER NO.. 4000-019-038-5200-00

DOC. CONTROL NO.: 4000-019-038-AAAK

cc Bruce Woods, RAP-WAM, U S. EPA, Region X  
Dena Hughes, Site Manager, WESTON, Seattle  
Kevin Mundell-Jackson, Database Management, WESTON

The quality assurance review of two sediment samples, laboratory batch K9805457, collected from the Duwamish River has been completed. The sediment samples were analyzed for organotins by Columbia Analytical Services of Kelso, Washington. Samples were analyzed by gas chromatography with an FPD detector. The samples were numbered:

98334029

98334030

### Data Qualifications

The following comments refer to the laboratory performance in meeting the quality control criteria described in the technical specifications of the laboratory subcontract. The review follows the format described in the *National Functional Guidelines for Organic Data Review* (EPA OSWER Directive 9240.1, February 1994), modified to include specific requirements of analytical methods.

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98-0627J 003  
DCN 4000-019-038-AAAK

17 November 1998  
Region X





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1. Timeliness

Holding time limits of 7 days for sample extraction and additional 7 days for analysis were established in the project Sampling and Analysis plan. All samples met holding time criteria.

2. Detection Limits—Acceptable

Instrument detection limits met project required quantitation limits.

3 Initial Calibration

A six-point initial calibration was performed prior to each analytical batch. The percent relative standard deviation for the initial calibration was within limits of less than 25 percent RSD

4. Continuing Calibrations

Continuing calibration check was performed after every 10 samples. All target analytes were within required limits for the continuing calibrations with the percent difference for a mid-range standard less than 25 percent.

5 Blanks

a) Laboratory Method Blanks

Laboratory method blank frequency criteria were met. No target analytes were reported in laboratory method blanks.

b) Field Blanks

No field blanks were associated with this SDG

6. Surrogate Compound Recovery

Surrogate recovery goals for tri-n-propyltin were established in the project Sampling and Analysis Plan at 60 to 120 percent for both sediment and porewater. Based on conversations with the laboratory an additional surrogate, triphenyltin was added and

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historical laboratory control chart limits were also used for data qualification. Laboratory limits are presented below:

Surrogate Compound	Sediment Limits	Porewater Limits
Tnpropyltin	20 - 195%	20 - 113%
Tripenyltin	20 - 175%	20 - 133%

Surrogate compound percent recovery met quality control criteria for all samples, with the exception of the following:

Sample	Surrogate	Percent Recovery
K980818-LCS (8/21/98)	Tripenyltin	59
K980818-LCS (9/21/98)	Tripenyltin	50
K980818-MB (9/21/98)	Tnpropyltin	51

Sample results are qualified as estimated (J) when both surrogate recoveries are outside project limits

#### 7. Laboratory Control Sample (LCS)

LCS recovery goals for tributyltin were established in the project Sampling and Analysis Plan at 60 to 130% for both sediment and porewater. Based on conversations with the laboratory, historical control chart limits of 20 to 138 percent for water and 20 to 164 percent for sediment were also used for data qualification.

All laboratory control sample percent recoveries met QC guidelines (P-project, L-laboratory), with the exception of the following

LCS	Analyte	Percent Recovery	QC Limit	Associated Samples
K980818-LCS (8/21/98)	n-Butyltin	20	60-130 (P) 20-164 (L)	98334029
K980818-LCS (9/21/98)	n-Butyltin	20	60-130 (P) 20-164 (L)	98334030

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Sample results for n-Butyltin were qualified as estimated (J) when LCS recoveries were outside project limits. Undetected results for n-Butyltin were qualified as estimated (UJ) when LCS recoveries were outside project limits.

#### 8. Matrix Spike/Matrix Spike Duplicate (MS/MSD) Analysis

The following matrix spike recovery goals were established in the project Sampling and Analysis Plan at for both sediment and porewater.

Analyte	% Recovery
Tributyltin	40 - 120%
Dibutyltin	30 - 120%
n-butyltin	10 - 120%

MS/MSD sample percent recoveries and relative percent differences (RPDs) met QC guidelines, with the exception of the following:

Analyte	MS %R	MSD %R	RPD	Associated Samples
n-Butyltin	155	91	30	98334029 98334030

Additionally, MS/MSD sample percent recoveries and relative percent differences (RPDs) for Tri-n-Butyltin and Di-n-Butyltin were not calculated due to high analyte concentrations in the sample. Results were not qualified solely on matrix spike/matrix spike duplicate results.

#### 9 Field Duplicate Analysis

No field duplicates were associated with this SDG.

#### 10 Sample Analysis

A cursory review of raw data was performed. All results were verified on a second, dissimilar, confirmation GC column. No unusual problems were noted. The case narrative noted that the high RPD value for di-n-butyltin in replicate analyses for sample 98334030 were possibly a result of incomplete derivatization. The sample and associated QC were



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rederivatized and reanalyzed, similar RPD results were obtained (107%). Based on variability seen in the replicate analyses, di-n-butyltin in sample 98334030 was qualified as estimated (J). The matrix spike and matrix spike duplicate recoveries for di-n-butyltin and tri-n-butyl tin in sample 98334029 were not calculated due to high analyte levels in the sample. The laboratory has begun spiking at higher levels in an effort to obtain useful matrix spike data. No other problems were noted.

#### 11. Laboratory Contact

No laboratory contact was required.

#### Data Assessment

Upon consideration of the data qualifications noted above, the data are ACCEPTABLE for use except where flagged with data qualifiers that modify the usefulness of the individual values.

#### Data Qualifiers

- U - The compound was analyzed for, but was not detected.
- UJ - The compound was analyzed for, but was not detected. The associated quantitation limit is an estimate because quality control criteria were not met.
- J - The analyte was positively identified, but the associated numerical value is an estimated quantity because quality control criteria were not met or because concentrations reported are less than the quantitation limit or lowest calibration standard.
- R - Quality control indicates that data are unusable (compound may or may not be present). Resampling and reanalysis are necessary for verification.
- N - Presumptive evidence of presence of material (tentative identification).

## COLUMBIA ANALYTICAL SERVICES, INC.

## Analytical Report

**Client:** Roy F Weston, Inc  
**Project:** Duwamish River/4000-027-001-2019-38  
**Sample Matrix:** Sediment

**Service Request:** K9805457  
**Date Collected:** 8/12/98  
**Date Received:** 8/13/98

## Butyltins

Sample Name 98334029 Units ug/Kg (ppb)  
Lab Code K9805457-002 Basis Dry  
Test Notes D

Analyte	Prep Method	Analysis Method	MRL	Dilution Factor	Date Extracted	Date Analyzed	Result	Result Notes
Tetra-n-butyltin	Method	Butyltins	5	5	8/18/98	8/22/98	ND	
Tri-n-butyltin	Method	Butyltins	5	5	8/18/98	8/22/98	54	
Di-n-butyltin	Method	Butyltins	5	5	8/18/98	8/22/98	14	
n-Butyltin	Method	Butyltins	5	5	8/18/98	8/22/98	10	5

7/29/98 10/28/98

D The MRL is elevated because of matrix interferences and because the sample required diluting

Approved By

*TG*

Date

9/22/98

1S22/020597p

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## Butyltins

**Sample Name** 98334030 **Units** ug/Kg (ppb)  
**Lab Code** K9805457-003 **Basis** Dry  
**Test Notes** D

Analyte	Prep Method	Analysis Method	MRL	Dilution Factor	Date Extracted	Date Analyzed	Result	Result Notes
Tetra-n-butyltin	Method	Butyltins	5	5	8/18/98	9/22/98	6	
Tri-n-butyltin	Method	Butyltins	5	5	8/18/98	9/22/98	190	
Di-n-butyltin	Method	Butyltins	5	5	8/18/98	9/22/98	21	J
n-Butyltin	Method	Butyltins	5	5	8/18/98	9/22/98	40	J

MGT 10/28/98

D

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Approved By



Date

9-22-98

1522/020597p